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Investigation of Physical-Mechanical and Thermos Gravimetric (TGA) Characteristics of the Combined Effect of Boron Nitride (BN) and Aluminum Oxide (Al_2O_3) in Polyethylene Terephthalate (PET) Matrix.

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Abstract:

Composite materials, particularly those incorporating polymers, have gained significant attention in recent years due to their unique combination of properties that can be tailored for specific applications. This research investigates the physical and mechanical characteristics of a composite material composed of matrix Polyethylene Terephthalate (PET), and reinforcement of Boron Nitride (BN), and Aluminum Oxide (Al_2O_3). The incorporation of these distinct components aims to synergize their individual properties, resulting in a composite material with enhanced performance for various engineering applications. The manufacturing process involved melt blending PET with varying concentrations of BN and Al_2O_3 by injection molding to produce test specimens as per ASTM standards. The physical and mechanical properties of the composite, including density, thermal conductivity, and morphological features, were analyzed through techniques such as optical microscopy and density measurements. Mechanical properties, such as tensile strength, percentage elongation, and impact resistance, were analyzed. Tensile testing revealed the effects of filler content on the strength, elasticity, and elongation of the material, Impact testing assessed the composite's ability to withstand sudden and dynamic loads. Furthermore, the thermal stability and degradation behavior of the PET+BN+Al_2O_3 composite were investigated through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). These thermal analyses aimed to elucidate the material's performance under elevated temperatures and its potential applications in environments with varying thermal conditions. The results of this study contribute valuable insights into the synergistic effects of combining PET with BN and Al_2O_3, providing a comprehensive understanding of the physical and mechanical properties of the composite material. The findings may pave the way for the development of advanced materials with tailored properties for specific engineering applications, such as lightweight structural components, heat-resistant materials, or impact-resistant surfaces.

1. Introduction:

In today’s modern day, every researcher and manufacturers are looking for a material with the high strength-to-weight ratio for numerous applications. Polymer composites are known for their low density, resulting in lightweight materials. This characteristic is particularly advantageous in industries like aerospace and automotive, where reducing weight can lead to improved fuel efficiency and overall performance. Polymer composites hold significant importance in various industries and applications due to their unique combination of properties. Polyethylene terephthalate (PET) is a thermoplastic polymer that is widely used in various applications like packaging, textiles, and engineering plastics [1-5]. The versatility of polymer composites allows for complex shapes and designs. This flexibility in design is particularly useful in industries where intricate structures are required, such as in automotive components and consumer goods. PET Polymer composites often exhibit improved mechanical properties, such as higher strength, stiffness, and toughness, when reinforced with fillers like boron nitride (BN) [6-9] and aluminum oxide (Al_2O_3) [10-14], compared to the individual components. This makes them valuable for structural applications where traditional polymers may fall short. PET can be prone to brittleness, especially at low temperatures. The addition of BN and Al_2O_3 can help reduce brittleness and improve the material's performance in a wider range of temperatures [15-18].

2. Materials and methods of Experimental work

2.1. PET/ BN/Al_2O_3

Polyethylene Terephthalate (PET): PET as a thermoplastic polymer widely used in various applications such as packaging, textiles, and engineering plastics. In addition, excellent in mechanical properties, chemical resistance, and low cost, making it a popular choice in composite materials. Accucomp PET008L grade PET density of 1.38 g/cm^3 99% commercial purity was used.

Boron Nitride (BN): BN as a ceramic material with high thermal conductivity, electrical insulation, and chemical stability. Emphasize its unique hexagonal structure and ability to enhance the thermal and mechanical properties of composites. BN (purity> 99 %) 2.1 g/cm^3 density

Aluminum Oxide (Al_2O_3): Aluminum oxide as a ceramic material known for its high hardness, excellent wear resistance, and thermal stability. It has potential contributions to improving the mechanical and wear properties of composite materials. Al_2O_3 of (purity: > 99 %) 3.95 g/cm^3 were obtained from M/s Nanoshell LLC and Intelligent Materials Pvt. Ltd, India.

2.2. Preparation of PET/BN/Al_2O_3 composites

In a lab-developed ball milling setup, the powdered PET polymer and BN+Al_2O_3 particles were combined and ball milled at room temperature in the appropriate ratios according to the calculated weight percentage (0 – 40%) using equation 1. In the ball mill, zirconia balls were used to mix PET powder with BN and Al_2O_3 powders in a 10:1 ratio. For four hours, in dry conditions, the ball mill was run at 60 rpm. BN and Al_2O_3 micro size powder was added to the PET matrix at weight percentages of 0, 5, 10, 15, 20, 30, and 40%. By stopping the machine for fifteen minutes every hour during the ball milling process, the mixture's temperature is kept at room temperature. The mixed powders dried in an oven (INLAB Equipment Pvt. Ltd., Chennai, India) at 180 °C to make sure all moisture was removed from composite mixture. The dried composite powder was taken and processed in the injection molding machine at 200 bar injection pressure as shown in Fig. 1 depicts the schematic of the injection molding machine. Compared to compression or hot compacting molding processes, the advantages of the injection molding process include process automation, low cost, better-quality contours and details, economical manufacturing costs, larger volume components, and mass production. The addition of BN and Al_2O_3 can enhance the process ability of PET during manufacturing processes such as extrusion and injection molding. This can lead to better-formed products with improved mechanical properties. [10–12].

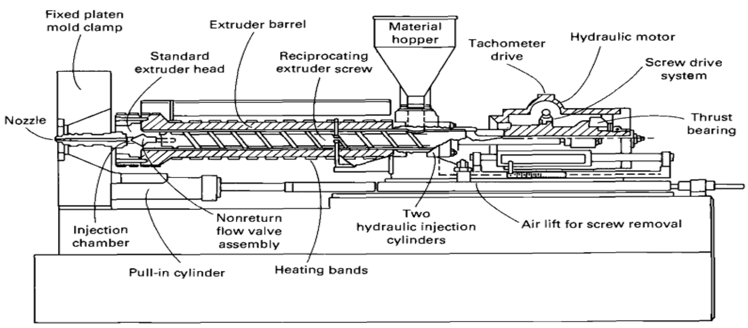

Figure 1. Schematic diagram of molding machine

PET polymer has a melting temperature of 260 ^{0}C, which was reached in three stages in the barrel heaters. The composite material softens between 200 and 250 ^{0}C due to heat transfer from the barrel wall heaters, and as the lead screw rotates, it applies high shear forces to the powder and moves it into the die cavity. At 290 ^{0}C, the powder completely melts and was injected under high pressure of 200 bar through runner and gates into the mould die. The die mould was then allowed to cool to room temperature in order to solidify. Equation 1 used to prepare the specimens as indicated in Table 1 for various weight percentages. The specimens were fabricated in accordance with ASTM D256 for impact testing, D628 for tensile testing [19-20].

Table 1. Specimen codes and compositions.

Specimen	code	PET-0	PET--5	PET-10	PET-15	PET-20	PET-30	PET-40
BN	**Wt. %**	0	2.5	5	7.5	10	15	20
Al_2O_3	**Wt. %**	0	2.5	5	7.5	10	15	20
$BN+Al_2O_3$	**Wt. %**	0	5	10	15	20	30	40
	Vol.%	0	2.61	5.43	8.47	11.78	19.31	28.4

Determination of wt. % and vol. % of PET/BN/Al_2O_3 composite, The equivalence vol. fraction (V_f) of wt. fraction (W_f) of BN and Al_2O_3 were calculated by using equations:

For pure PET, required weight for sample preparation was calculated by

Mass (m) = Volume (V) * Density (ρ), gms (Eqn. 1)

V = volume of sample, cm^3, ρ = density, g/cm^3, m = mass, g

$$V_f = \text{Volume fraction} = \left[\frac{W_f}{(W_f + (1-W_f))\left(\frac{\rho_f}{\rho_m}\right)}\right] \quad \text{(Eqn. 2)}$$

Where, W_f = wt. fraction of reinforcement,

ρ_{f1} = density BN (2.1 g/cm^3), ρ_{f2} = Al_2O_3 (3.8 g/cm^3), ρ_m= density PET (1.38 g/cm^3).

Table 1. Shows the W_f Composition and equivalent V_f in percent. Theoretical density $\rho c_{(th)}$, of PET/BN/Al_2O_3 composite and weight of matrix and filler required for sample preparation of PET/BN/Al_2O_3 composite are calculated by "Rule of Mixture (ROM)" [10] by using Archimedes principle experimental density was calculated [10-12]. The average of 3 readings were considered for calculations, specimen is weighed in air and later when immersed in medium and its weight is recorded.

2.3. Characterization

The rule of mixtures (ROM) was used to calculate the theoretical densities of the PET/BN/Al_2O_3 composite [10-12]. The PET matrix density was 1.38 g/cm^3, and the BN reinforcement density was 2.1 g/cm^3 and Al_2O_3 is 3.8 g/cm^3. It was assumed that during the specimen fabrication process, there was no loss and no cavities formed. The specimens weighed using an AFCOSET model ER200A high precision electronic balance. PET/BN/Al_2O_3 composite microhardness was measured at 100 g load and 10 s dwell time using the Model-MVH–S-AUTO-Z from Metatech Industries in Pune, India. PET/BN composites' microhardness were calculated using an average of five readings.

Using a UTM machine (Model: UNITEK-16100-STS), tensile tests are performed until the specimen breaks. The test results generally suggest or predict the material behaviors and responses under other different loadings, which helps to select composite for a suitable application. A speed rate of 5 mm/min was used to apply a load, and the percent elongation and applied forces were recorded. Tensile strength, elongation, and percentage area reduction are among the characteristics of a tensile test that can be measured directly. Fine Testing Machine (FTM), Model

FIT-14, Miraj, India, used for the impact test results for PET/BN/Al_2O_3 composites were acquired. The impact toughness of a material concerning its cross-sectional area is determined by the energy (J) absorbed by the specimen before fracture. The energy absorbed before the specimen's cross-sectional area fractures determines the impact strength of PET/BN/Al_2O_3 material.

3. Results and discussions

Physical Properties

3.1. Density:

The density of the composite is influenced by the addition of fillers. Typically, the density increases with the addition of BN and Al_2O_3. The density of a composite material like PET/BN/Al_2O_3 depends on the proportions of each component in the mixture. Fig. 2 displays the theoretical and experimental densities of PET/BN/Al_2O_3. The composite densities increase linearly with an increase in the BN and Al_2O_3 percent. The results demonstrate appropriate ball mill blending and high-quality samples of specimens free of defects during the injection molding process. The theoretical and experimental densities of PET/BN/Al_2O_3 are more similar to each other at lower volume percentages (PET-0, PET-5, PET-10, and PET-15) and at upper volume % (PET-20, PET-30, and PET-40), where porosity was observed in greater amounts due to the addition of BN/Al_2O_3 percent. These results differ from linearity. The porosity verifies that when BN and Al_2O_3 weight percentage is added, the interparticle space shrinks, leading to larger agglomerations of BN and Al_2O_3 elements. It becomes more difficult to control the uniform distribution of BN and Al_2O_3 particles in the molten polymer at the machine barrel, which causes agglomeration, which is confirmed by optical microscopy and results in porosity. When compared to theoretical density, the PET-40 composites showed a maximum porosity of 4.05%. "Rule of Mixture (ROM)" is used to compute the theoretical density and weight of the matrix and filler needed to prepare a PET/BN/Al_2O_3 composite sample.

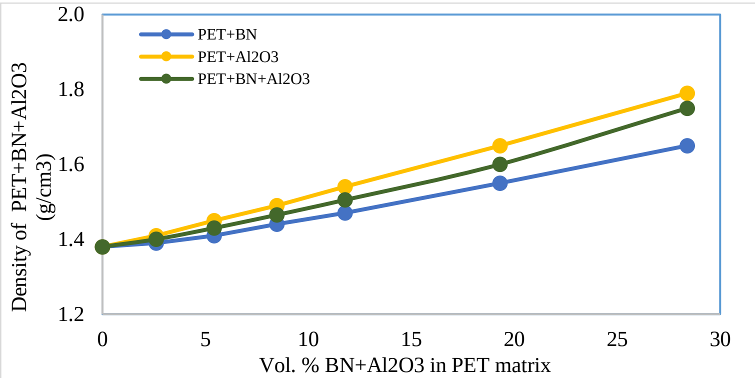

Fig. 3.1, Densities of PET+BN, PET+ Al2O3 and PET-BN+ Al2O3 Composite

3.2 Micro hardness:

The microhardness of a material is a measure of its resistance to deformation or indentation and is influenced by the composition and structure of the filler material. The addition of reinforcement BN/Al_2O_3 fillers has increased the hardness of the pure PET in the composites. The maximum microhardness values of the PET-40 composite are magnified by times of pure PET polymer matrix. Vickers indenter penetration is resisted by the homogenous dispersion of BN/Al_2O_3 particles in the matrix, leading to significantly enhanced composite microhardness this is due to more crystal nucleation and a local polymer chain conformation present in the areas close to the BN/Al_2O_3 reinforcing particles than in the areas farther away. The interactions and bonding between the components can affect the overall mechanical properties. The results show Fig. 3.2 The combined addition of BN+Al_2O_3 shows better results compared to the individual addition of BN and Al_2O_3, this is due to good mechanical bonding and resistance to indentation leading the good hardness. The addition of BN can potentially improve the microhardness of PET.

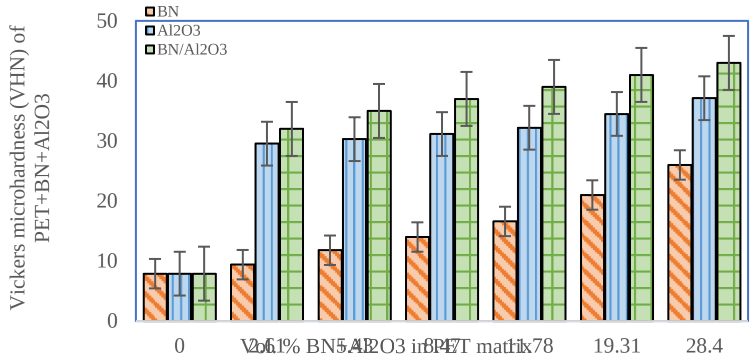

Fig. 3.2, Microhardness of PET+BN, PET+ Al2O3 and PET-BN+ Al2O3 Composite

3.3 Impact strength:

Impact strength refers to the ability of a material to withstand sudden force or shock loading without fracturing or breaking. It is a measure of the energy absorbed by a material during impact before it fractures. Both BN and Al_2O_3 act as reinforcing agents in the polymer matrix. The impact energy and strength is influenced by the type and concentration of fillers. While some fillers may enhance impact resistance, others might make the material more brittle. Achieving a balance between stiffness and toughness is crucial. Both BN and Al_2O_3 materials have unique properties that can enhance the overall performance of the composite.

Figure 3.3, shows the results of the impact test results of PET+BN, PET+Al_2O_3 and combined effect of PET+BN+Al_2O_3 composites at room temperature. Comparing pure PET matrix and high concentration PET+BN+Al_2O_3 powder, the fracture energy and impact strength of increases and more energy is absorbed. As the amount (weight percent) of both fillers BN + Al_2O_3 particles per unit volume increases (i.e. the impact absorbed energy also increases), further crack propagation in the material are blocked. Since the contact area of PET decreases with increasing weight fraction, the wettability of the BN+Al_2O_3 particles increases and the energy transfer from the matrix to the BN+Al_2O_3 reinforcement increases, resulting in an increase in fracture energy and impact strength. Due to agglomeration of BN+Al_2O_3 reinforcement with higher weight percent, the grain boundaries of PET+BN+ Al_2O_3 composites are enlarged, resulting in an increase in the impact energy and strength. BN is known for its high thermal conductivity, it can help in better heat dissipation. This can prevent localized overheating and degradation of the material due to impact [10]. By improving the impact strength of PET, it may be possible to design lighter-weight products without sacrificing strength. This can be particularly beneficial in industries where weight is a critical factor, such as automotive and aerospace [11].

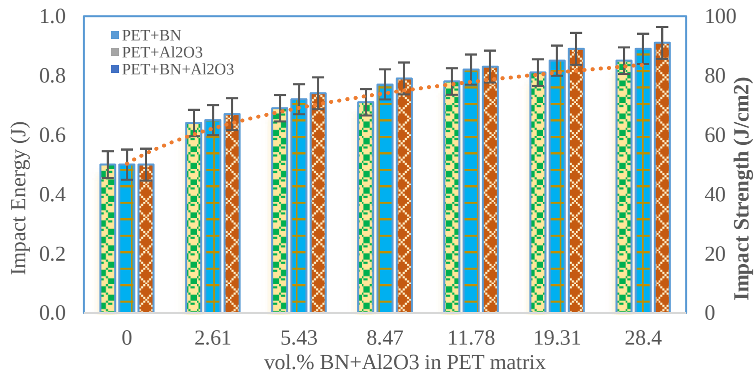

Fig. 3.3, Impact Energy and strength of PET+BN, PET+ Al_2O_3 and PET-BN+ Al_2O_3 Composite

3.4 Tensile strength:

The tensile strength of PET (Polyethylene terephthalate) reinforced with materials like Boron Nitride (BN) and Aluminum Oxide (Al_2O_3) depends on various factors such as the manufacturing process, the ratio of reinforcement materials to PET, and the dispersion of reinforcement materials within the PET matrix. However, generally speaking, both BN and Al_2O_3 are known to enhance the mechanical properties of polymers like PET. Fig. 3.4 shows the tensile strength of PET reinforced with BN and Al_2O_3. Boron Nitride (BN) can improve its tensile strength, particularly in applications requiring high-temperature resistance. Aluminum Oxide (Al_2O_3), is also used as a reinforcement material in polymer composites due to its high hardness, stiffness, and thermal stability. Al_2O_3 can enhance its mechanical properties, including tensile strength and modulus. The actual improvement in tensile strength would depend on several factors including the concentration of BN or Al_2O_3 in the composite, the method of dispersion, and any surface treatment applied to the reinforcement materials to enhance bonding with the PET matrix.

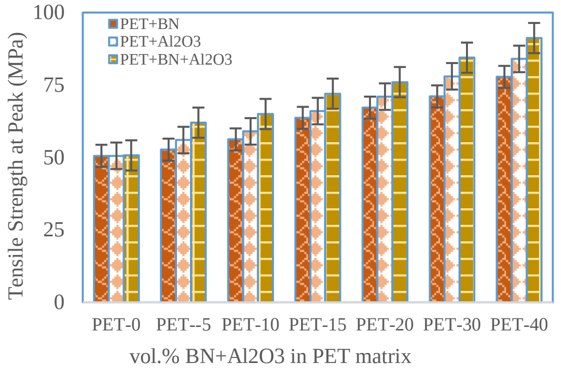

Fig. 3.4, Tensile strength at Peak of PET+BN, PET+ Al_2O_3 and PET-BN+ Al_2O_3 Composite

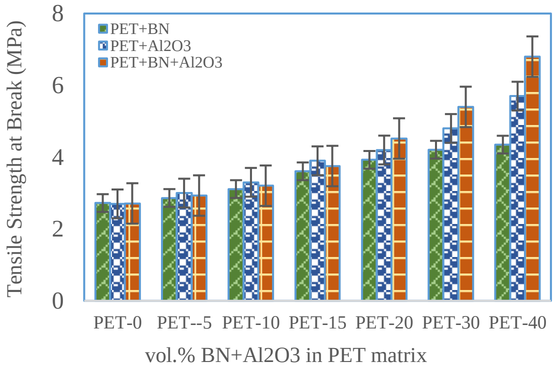

Fig. 3.5, Tensile strength at break of PET+BN, PET+ Al_2O_3 and PET-BN+ Al_2O_3 Composite

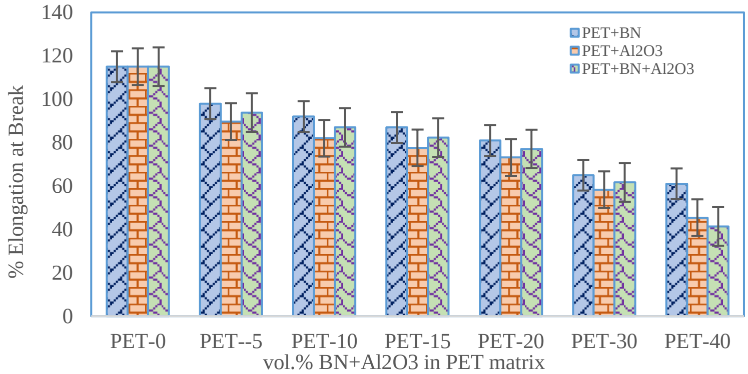

Fig. 3.6, Percent elongation at break of PET+BN, PET+Al_2O_3 and PET-BN+ Al_2O_3 Composite

3.5 Coefficient of Thermal Expansion

The coefficient of thermal expansion (CTE) is a material property that quantifies how much a material expands or contracts with temperature changes. The coefficient of thermal expansion (CTE) of a composite material such as PET (Polyethylene terephthalate) reinforced with Boron Nitride (BN) and Aluminum Oxide (Al_2O_3) can be influenced by various factors including the type and amount of reinforcement, processing conditions, and the matrix material itself. Generally, adding reinforcing materials can alter the CTE of the composite compared to the pure matrix material. Fig. 3.7 shows the effect of Boron Nitride (BN) and Aluminum Oxide (Al_2O_3) Boron Nitride (BN) is known for its relatively low coefficient of thermal expansion, making it suitable for thermal management applications. Aluminum Oxide (Al_2O_3) also tends to have a lower CTE compared to many polymers. However, to provide specific values for the CTE of PET reinforced with BN and Al_2O_3, experimental data are shown in Fig. 3.7. These values can vary depending on the specific composition and processing conditions of the composite material.

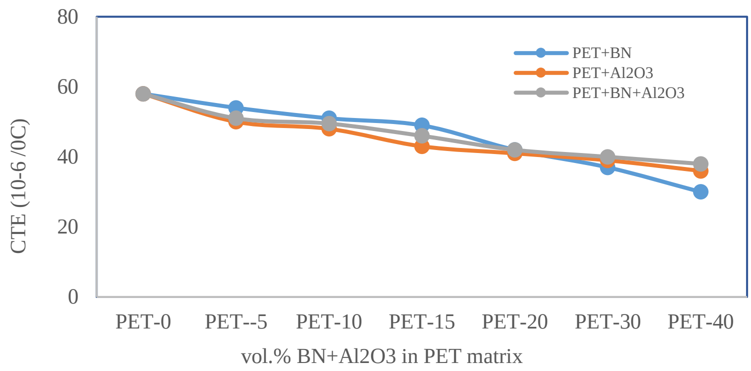

Fig. 3.7, Coefficient of Thermal Expansion (CTE) of PET+BN, PET+Al_2O_3 and PET-BN+ Al_2O_3 Composite

It's important to note that these values are approximate and can vary depending on factors such as the specific form of the material (e.g., single crystal, polycrystalline), impurities, and the temperature range of interest.

3.6 Thermo Gravimetric analysis (TGA)

Thermogravimetric analysis (TGA) of PET (polyethylene terephthalate) reinforced with fillers like boron nitride (BN) and aluminum oxide (Al_2O_3), provides insights into their thermal stability and degradation behavior. At the beginning of the TGA, you'll typically observe a slight weight loss due to the evaporation of any moisture or volatile compounds present in the sample. This is usually seen at lower temperatures as shown in Fig. 3.8

Polymer Decomposition increases with temperature rises, PET will start to degrade thermally. The TGA curve will show a more significant weight loss as the polymer chains break down into smaller molecules and eventually volatilize. This degradation usually occurs in a single step for PET, typically around 400-500°C. The presence of fillers like BN and Al_2O_3 can influence the thermal degradation behavior of the composite. Fillers can act as thermal stabilizers, affecting the onset temperature of decomposition or altering the degradation mechanism. The TGA curve may exhibit shifts in the onset temperature or changes in the rate of weight loss compared to pure PET.

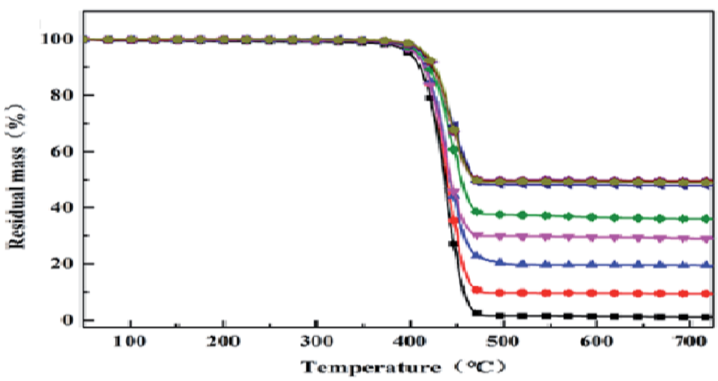

Fig. 3.8, Thermo Gravimetric analysis (TGA) of PET-BN+ Al_2O_3 Composite

TGA can be used to compare the thermal stability of different composite formulations with varying filler content or types. By comparing the onset temperatures of degradation, rates of weight loss, and residue amounts, these results show the effectiveness of fillers in enhancing the thermal properties of the polymer composite.

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4. Conclusion:

Reinforcing PET polymer with BN and Al_2O_3 can contribute to improved overall mechanical properties, including tensile strength and modulus of elasticity. The PET-(BN+Al_2O_3) composites contributed up to 40% of pure PET. The density of the composite increases with an increase in BN+Al_2O_3 percent. Theoretical and experimental densities are similar at lower volume percent (5-20 vol. %), as confirmed by optical microscopy, whereas at higher volume percent (30-40 vol. %) 4.05 % porosity was reported due to the agglomeration of BN+Al_2O_3 particles producing a three-dimensional network structure, as revealed by optical microscopy. Compared to virgin PET polymer microhardness was enhanced by almost 81% and in line with theoretical hardness, BN and Al_2O_3 are hard materials, and their addition can increase the overall hardness of the composite. Higher hardness often correlates with better resistance to impact and wear. Impact energy and impact strength were about 45% higher, as tensile strength showed 45% which are significant improvement compared to virgin PET polymer. The results validate this PET-(BN+Al_2O_3) composites exhibit good mechanical properties, lower Coefficient of thermal expansion, and stable up to 400^0C temperature. The addition of BN and Al_2O_3 can improve the chemical resistance of the polymer. This is valuable in environments where exposure to harsh chemicals may compromise the integrity of the material. The enhanced hardness can contribute to better resistance against chemical attacks, thus these composites have vast potential in automotive, robotic, and food-processing material applications. PET/BN/Al_2O_3 composite could be beneficial, such as in electronic packaging, thermal management, and structural components requiring a balance of mechanical and thermal properties.

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